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WHOLE PLANT UTILIZATION OF SUNFLOWERS AS A RENEWABLE SOURCE OF STRATEGIC MATERIALS (RUBBER)

Final Report

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In an attempt to discover and develop new domestic sources of natural rubber (NR), 143 populations of 47 species of sunflowers were collected and analyzed for their content of NR. Yields of crude NR obtained ranged from traces to 2.15% of the dry weight of the plant material. These quantities of NR are significantly lower (by approximately one order of magnitude) than those obtainable from domestic guayule plants. In addition, samples of sunflower NR were examined using chromatographic and spectroscopic techniques and were found to have low molecular weight elastomeric properties unsuitable for use in

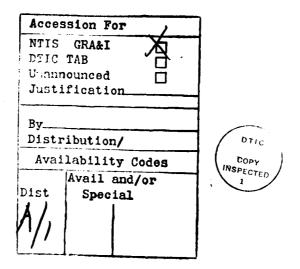
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traditional <u>Hevea</u> latex or guayule NR applications. Thus, because of low yields, low NR molecular weights and quality, and relatively narrow genetic diversity in sunflower NR production characteristics, it is highly unlikely that sunflowers could serve as a domestic source of NR in the foreseeable future.



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BACKGROUND

Natural rubber (NR) is, chemically, cis-1,4-polyisoprene. Although synthetics such as silicone rubber, polyurethane, fluoroelastomers, and petroleum-derived BR are more widely publicized among rubber materials, NR (currently derived from the latex of Hevea brasiliensis) has many advantages over these. Natural rubber is stronger and has better abrasion resistance than silicone, has more resilience than polyurethane, and has better subzero performance than fluoroelastomers (Wolfe, 1979). In addition, NR has excellent resistance to impact, tear, and heat build-up, and has good tensile strength, green strength, flexibility, aging stability and building tack (Larsen, 1979). In general, NR offers one of the best blends of engineering properties available in rubber materials, and cannot easily be replaced by synthetics. It is safe to state that, overall, no man-made rubber has ever performed as well as NR (Imle, 1978).

Natural rubber is a critical strategic material, and is thus an essential industrial material of vital importance to the U.S. economy and security. NR is essential in the manufacture of aircraft tires, radial tires, rubber surgical supplies, and many other industrial products. For example, aircraft tires use from 90 to 100% NR, while truck and bus tires use almost 50% NR. Auto radial tires contain approximately 14-15% NR. Such uses account for more than 80% of the NR imported into the U.S. annually (Wolfe, 1979).

Although NR is available today at relatively low cost, the U.S. is totally dependent upon foreign supplies of this strategic material because no current domestic sources of NR exist. This places the U.S. in a potentially vulnerable position, since the availability of NR is subject to the political and military stability of the producing countries, and their favoritism towards the U.S. The key role that this material plays in our economy and the magnitude of our dependence on foreign sources is highlighted by noting that the U.S. is the world's largest consumer of NR, importing from 20% to 25% of the world's production annually. NR is also the second largest volume elastomer used in the U.S., accounting for almost 25% of the annual domestic rubber consumption (Wolfe, 1979).

There is an obvious need to develop stable and renewable domestic sources of natural rubber, both to meet future increasing demands, and also to ensure stable supplies of this material in times of national emergency. Although a number of plant species produce NR (Bruehrer and Benson, 1945; Polhamus, 1957, 1967; Buchanan et al., 1978a,b; Swanson et al., 1979), extensive screening and economic (agronomic) considerations have permitted selection of a few promising species which currently require further development towards domestication and commercialization. These include guayule, sunflowers, and rubber rabbitbrush. All of these rubber-producing species belong to the same plant family, the Compositae (Asteraceae). This is the largest and most advanced family of flowering plants.

It should be noted that considerable research has been conducted on guayule and that the properties of its rubber have been found to be very good. However, guayule is not tolerant to frost and therefore can only be grown in a relatively small area of the United States (frost-free belt). The frost-free belt is (and will likely always be) under intense pressure for growing citrus

and other food crops. In addition, this area is generally very arid and irrigation water must be applied. These resources are obviously limited.

A tremendous opportunity exists in the United States for the introduction of a domestic rubber crop into American agriculture. It is estimated that about 110 million acres of available cropland are not cropped each year and that an additional 90 million acres could be developed, not including desert and marshlands. These 200 million acres compare favorably with the 350 million acres cropped annually (Princen, 1982).

Sunflowers (Helianthus species) are some of the most widely distributed and adapted species in the United States. Helianthus annuus has developed into a major agricultural crop (4.5 million acres) prized for its excellent quality seed oil (composed largely of triglycerides) (Beard, 1981). There is currently considerable interest in the direct use of sunflower oil as a diesel fuel substitute (Morgan and Shultz, 1981; Hoffman et al., 1980) and in catalytic conversion to other fuels (Weisz et al., 1979; Haag et al., 1980). However, little attention has been given to the potential utilization of whole plant sunflower extracts as a source of natural rubber (Swanson et al., 1979; Stipanovic et al., 1980, 1982; Adams and Seiler, 1984). With the increased demand for natural rubber in radial tires plus the fact that rubber is essential to our country's defense, there is a need to develop a domestic source of this commodity. The aforementioned screening studies have identified several sunflower species with potential for commercial development. This opportunity is further enhanced by the wealth of data available on the genetics, crossability and agronomics of Helianthus. Not only are there approximately 100 species of Helianthus, but three closely related genera (Phoebanthus, Tithonia, and Viguiera) which contain natural rubber. It is important to note that added-value products can turn an uneconomic crop into a very valuable one. This is an important aspect in getting a strategic crop started without incurring large government subsidies. The extracted residue of sunflowers, for example, may be utilized as cattle feed.

The taxonomy and evolution of Helianthus have been studied extensively by Charles Heiser (Heiser et al., 1969). About 100 species of Helianthus are known, 50 of them native to North America and 15 to South America (Beard, 1981; Heiser et al., 1969). The genus includes both annuals and perennials, with considerable natural hybridization. Most species have a chromosome number of n=17. Tithonia (Mexican sunflower) is a closely related genus with 11 species, four of which have a haploid number of 17. Tithonia is a popular ornamental in California and certain species have been found to contain over 2% natural rubber (Stipanovic et al., 1982). The genus Viguiera (Heliomeris by some authors) is even more closely related to Helianthus. Eight of the 17 species of Viguiera (Goldeneye) have a haploid chromosome number of n=17. Viguiera commonly grows in the harsh environs of Baja California, but some species are found as far north as Montana. Phoebanthus is also closely related to Helianthus, and Heiser et al. (1969) suggest that they be classified together. Phoebanthus comprises two species, both endemic to Florida. One is a diploid; the other is a tetraploid. Both have a basic chromosome number of n=17. No reports of attempted hybridization with Helianthus are known (Heiser et al., 1969).

A. Natural rubber. Although sunflowers have been little screened for

natural rubber, Swanson et al. (1979) reported that Helianthys hirsutus contains natural rubber with a molecular weight of 2.79 x 10 and a polydispersity factor (3.1) which indicates a potential as a commercial source of natural rubber. Other references to rubber in sunflowers are scattered throughout the literature, but the first extensive studies of this genus were conducted by Stipanovic et al. (1980, 1982). The 53 Helianthus taxa they screened contained from a trace of rubber to 1.89% in the leaves. While most species sampled were growing in a common garden, others were sampled from the wild. Sampling of both stems and leaves indicates that the rubber is found mostly in the leaves rather than in the stems, in contrast to guayule (Parthenium argentatum).

Helianthus annuus originating from Winton, Oklahoma, had 1.45% rubber (Stipanovic et al., 1982). The same species from Tucson, Arizona contained 0.55% NR (Bruehrer and Benson, 1945) and a sample collected in Ontario, Canada by Minshall (1957) contained only 0.26% rubber. This could be evidence of environmental effects or of genetic variation within species (infraspecific variation), and points out the need to do evaluations in a common environment in order to make direct comparisons and to evaluate genetic versus environmental variability. Stipanovic et al. (1982) found that Tithonia rotundifolia, a Mexican ornamental sunflower, had over 2% rubber in the leaves.

- B. Animal feed. Extracted sunflower residue should make an excellent high quality animal feed. Sunflowers have been used extensively on a whole-plant basis as an animal forage. Sunflowers were reintroduced to the U.S. from Russia as a silage crop recommended for planting on the fringes of the corn belt where they would be more productive than corn. Marx (1977) compared sunflower silage with alfalfa haylage, and found the sunflower product to contain 11% crude protein, 33.5% crude fiber, 7% ether extract, and 9.5% ash, of which 0.83% was calcium and 0.32% phosphorus. In feeding trials, sunflower silage was 92% as good as alfalfa haylage. A preliminary screening of cyclohexane/methanol-extracted residues of 39 Helianthus taxa identified accessions with up to 20% protein.
- C. Agronomics. A plant is of little use if it cannot be grown. Sunflowers are found in virtually all parts of the U.S. and are cultivated on every major continent. Many of the cultural practices used for producing a seed crop can be used to produce a chemical crop. Several taxa are desert species and are found growing in extremely harsh environments. Insect and disease resistance are being improved in the domestic hybrid through hybridization with wild species.
- D. Breeding and Selection. Sunflowers are easily cross-bred, and hybridize so frequently in the wild that their taxonomic classification is difficult (Heiser et al., 1969). Due to the several sources of male cytoplasmic sterility available, they make an excellent hybrid. They are highly self-sterile, and are hybridized by hymenopterous pollinators, principally honey bees. The numerous species and related genera with the same chromosome number should provide an extremely wide germplasm pool for future selection and genetic improvement.

RESULTS

Task 1. The objective was to survey additional populations of the nine most promising taxa of Helianthus previously identified. The following table lists the taxa, soil type, locations, and the number of populations collected in completion of this task. In addition, the % NR content (on a dry weight basis) is listed for each taxon.

Table 1.

Taxa	Number of Populations	Location	Soil Type	% NR
H. agrestis	4	FL	Damp mucks	0.75-2.15
H. annuus	15	UT, AZ	Variable	0.04-0.24
H. anomalus	1	UT	Dry sands	0.02
H. argophyllus	0		•	
H. arizonensis	1	TX	Dry sands	0.74
H. ciliaris	2	NM, TX	Variable	0.53-1.50
H. nuttallii	7	ID, TX	Variable	0.03-0.62
H. praecox hirtus	4	ΤΧ	Sands	0.12-0.26
H. resinosus	5	NC	Variable	0.26-1.15

Task 2. Screen 24 Helianthus taxa not grown at the USDA/SEA facility at Bushland, Texas. As many of these taxa as feasible were to be collected (5 populations/taxa, 10 plants each) for analysis. The 24 taxa are listed in Table 2, accompanied by the number of populations collected and their % NR content on a dry weight basis. Six of these taxa were found to be endangered or rare, viz, H. carnosus, H. debilis spp. vestitus, H. exilis, H. gracilentus, H. praecox spp. praecox, and H. schweinitzii. H. carnosus is rare and unusual, occurring only in wet sandy soils in northeast Florida. debilis spp. vestitus is limited to sandy soils along the central west coast of Florida. Heavy urbanization in this area has made this taxon very difficult to find. H. exilis is considered by some authors to be a synonym of H. bolanderi, and is endangered, occurring only in serpentine soils near Davis, California. H. gracilentus, while not endangered, occurs only along the coast of southern California and into Mexico. H. niveus spp. tephrode occurs mostly in Mexico, extending just north into Imperial County of California and the western edge of Arizona. A single population was located just east of Yuma, Arizona. H. schweinitzii occurs in small numbers in just a few sites in North and South Carolina. The fact that it has not been collected for several years indicates that it is endangered. H. praecox spp. praecox occurs only on Galveston Island and on the adjacent mainland.

Table 2. Taxa of Helianthus not previously screened.

	Number of			
Taxon	Populations Collected	Location	Soil Type	% NR
H. atrorubens	5	GA, NC	Variable	0.10-0.32
H. bolanderi	Ö	,		
H. carnosus	Ō			
H. cusickii	Ö			
H. debilis	6	FL, TX	Moist sands	0.55-0.79
cucumerifo	lius 4	SC, TX	Moist sands	
silvestris	2	TX	Sands	0.24-0.39
tardifloru		TX	Sands	0.67
vestitus				
H. decapetalus	- 0 5 . 1	NC, TX	Loams	0.14-1.46
H. eggertii		TX	Sands	0.65
H. exilis	0			
H. floridanus	4	FL, GA	Moist sands	0.28-0.65
H. giganteus	4	NC, TX	Variable	0.27-0.87
H. gracilentus	. 0			
H. heterophyll	ūs 5	FL	Wet sands	0.16-0.58
H. longifolius	4	AL	Variable	0.02-0.10
H. niveus	•			
canescens	7	TX	Sands	0.04-0.33
niveus	3 1	Baja, Mex.	Dry sands	0.21-0.44
tephrodes		AZ	Dry sands	0.10
H. nuttallii	7	AZ, UT,	-	
_		ID, Mex.	Moist sands	0.03-0.62
rydbergii	0			
H. praecox				
praecox	0			
runyonii	1	TX	Loams	0.17
H. radula	- 4	FL	Wet sands	0.50-1.50
H. schweinitzi	<u>1</u> 0			

Task 3. Survey three genera closely related to Helianthus. The three genera, Tithonia, Viguiera and Phoebanthus are thought to be interfertile with Helianthus. There are 11 species of Tithonia, but only two, T. rotundifolia and T. therberii, occur in the U.S. The former is used as an ornamental and the latter occurs in Arizona. The rest are endemic to central and northern Mexico. Consequently, the only Tithonia species collected was a single population of T. rotundifolia, growing in captivity.

Most of the 17 species of <u>Viguiera</u> are also located in Mexico, particularly in Baja California. Many are large perennial shrubs, growing in harsh environs. We were fortunate in having collected 15 different taxa, including a number of the large perennial forms from Baja. We were not so fortunate in our search for another rare species of <u>Viguiera</u>, <u>V. ludens</u>. Endemic to Mexico, the only report of its existence in the U.S. was a single observation in 1977 at Lobo Flats, Texas, 19 miles east of VanHorn. A thorough search of the area yielded no <u>V. ludens</u>. This taxon was probably introduced from Mexico in a load of cotton seed, in 1976 or 1977, and has since been eradicated.

The genus Phoebanthus consists of the two species \underline{P} . $\underline{grandiflora}$ and \underline{P} . $\underline{tenuifolia}$, both of which are found primarily in Florida. Table 3 lists the related taxa collected along with the number of populations and general location, as well as their % NR content on a dry weight basis.

Thus, a total of 143 distinct populations of sunflowers were collected, including 107 populations of Helianthus (representing 28 distinct taxa), 8 populations of Phoebanthus (2 taxa), 1 population of Tithonia (1 taxon), and 27 populations of Viguiera (16 taxa). Therefore, a total of 47 sunflower taxa from 143 populations were collected and analyzed for their NR content.

Table 3. Related taxa collected.

	Number			
Taxa	of Populations	Location	Soil Type	% NR
Phoebanthus grandiflor	a 3	FL	Moist sands	0.00-0.12
P. tenuifolia	<u>a</u> 3 5	FL	Sands	0.01-0.16
Tithonia rotundifolia	1	TN	Variable	0.19
Viguiera deltoidea	2	Baja	Coastal sands	
V. deltoidea var.		•		
chenopodina	2	Baja	Roadside	0.35-1.02
V. deltoidea var.		•		
parishii	7	CA	Coastal sands	0.03
V. deltoidea var.				
tastensis	2	Baja	Roadside	0.74-1.76
V. dentata	2 2	TX, Mex.	Sands	0.06-0.14
V. laciniata	2	CA	Roadside	0.04-0.07
V. lanata	1	Baja	Variable	0.09
V. longifolia	1	TX	Variable	0.18
V. microphylla	1	Baja	Variable	0.16
V. multiflora	5	TX, UT	Variable	0.03-0.10
V. parishii	1	CA	Roadside	0.17
V. purissimae	1	Baja	Roadside	0.04-0.06
V. reticulata	1	CA	Variable	0.03
V. subincisa	1	Baja	Coastal sands	0.13
V. tomentosa	2	Baja	Roadside	0.18-0.25

In addition to the species populations collected, 120 pounds of Helianthus annuus were collected in an attempt to extract quantities of rubber for quality tests. H. annuus was selected because it occurs naturally in large, dense stands. The material was cut, air-dried and baled using conventional haying equipment, proving that should whole plant utilization of sunflowers ever prove economical, harvesting methods are readily available. The large, woody perennial species would require the use of copicing and chipping equipment such as that used in woody biomass processing.

Plant samples (aerial parts) were dried at 60°C for 48 hours and then ground to pass a 2 mm mesh. The dried and ground samples were then extracted first with acetone (deresinification) (20 hours) and then with hexane (20 hours) to obtain the NR. The presence of NR was confirmed in a number of the highest-yielding samples using infrared (IR) spectrophotometry, proton nuclear

magnetic resonance (NMR) spectroscopy, and gel permeation chromatography (GPC).

Yields of crude NR ranged from traces (or not detectable) to 2.15% of the dry biomass weight. Examination of the extracts of the highest-yielding plants by IR and NMR revealed, however, that not all of the extracts consisted of pure rubber. Several contained waxy and resinous components characterized as complex aliphatic-type esters. Many of the NR extracts, including the highest-yielding extract from Helianthus agrestis (2.15%) were oily gums, indicative of low molecular weight NR. Examination of a number of these extracts by GPC revealed molecular weight ranges from 20,000 to 256,000. Polydispersity ranged from 8.3 to 19.5. The NR extracts were typically greenish-straw-colored gums which rapidly oxidized in the presence of air if antioxidant was not added. When compared to Hevea and guayule NR, sunflower NR behaved more like an elastomer than a solid, high molecular weight NR.

Task 4. Bales totaling some 120 lbs. of Helianthus annuus were collected, dried, ground, and bulk extracted, yielding a bulk sample of sunflower NR (approx. 0.05% of the dry weight of the plant material). A very hard wax was also obtained during processing. A portion of the NR sample was refined by further deresinification and decolorization. IR and NMR revealed that the refined sample (straw-colored) was essentially pure NR (cis-1,4-polyisoprene). However, this material was not totally solid and behaved like rubber cement. Because of the method of extraction, it contained no dirt or protein, and the ash content was negligible. This purified NR sample contained no natural antioxidant (as does Hevea NR), and consequently readily oxidized upon exposure to air. This oxidation and decomposition could be readily followed by IR and NMR spectroscopy.

Examination of crude <u>Helianthus</u> hexane extracts and resin fractions (acetone extracts) by capillary gas chromatography/mass spectrometry (GC/MS) revealed that they consisted largely of diterpenoid acids such as ent-kaurenoic acid.

Task 5. Evaluate the domestication potential of Helianthus species in cultivation at Bushland. A site visitation was made to the wild sunflower collection maintained by the USDA/SEA at Bushland, Texas. While the collection contained only 13 taxa of interest, and disease and maturity information were not available, it was an opportunity to evaluate the agronomic potential of wild sunflowers growing in cultivation. The basic agronomic characteristics of the 13 taxa are listed in Table 4.

[able 4. Agronomic characteristics of 13 sunflower taxa grown in a common garden at Bushland, Texas.

<u>[axa</u>	Height (m)	Seed size (mm)	Dry wt. (g)	Branching <u>Pattern</u>
d. agrestis	1-2	2-3	25.2	above
H. arizonensis	.23	3	30.2	below
H. atrorubens	.5-2	3 3	22.3	above
H. ciliaris	up to .7	3	32.5	below
H. debilis				
silvestris	up to 2		26.1	above
tardiflorus	.0607		26.4	above
H. decapetalus	.6-2	3-3.5	19.5	entire stem
H. eggerttii	1-2	5 3	20.0	above
H. floridanus	1-2		20.7	above
H. giganteus	1.5-3	5	31.2	above
H. nuttallii	up to 3		43.8	above
H. praecox hirtus	.0506		23.2	entire stem
H. runyonii	.0506		34.6	entire stem

Productivity is an important consideration for any potential crop. While the percent natural rubber is an important yield component for sunflowers, total productivity is best estimated considering all the yield components, including plant size and number of plants which can be grown in a unit area. The final measurement of productivity would be pounds of usable rubber per acre/year.

Based on the data in Table 4, the two most promising biomass producers would be H. nuttallii and H. giganteus. These two were the tallest, and produced the most dry matter. Both are of the series Gigantei, and also have the largest seed. Seed size is of concern only from an agronomic viewpoint, the larger size being more desirable. Seed of most wild species exhibit a dormancy which requires a rather elaborate treatment to induce germination. While valuable in the wild, this characteristic would have to be bred out as part of the domestication process.

Conclusions

Our survey has shown that many species of sunflowers produce small amounts of natural rubber. However, this NR is produced in too small a quantity (generally less than 2% of the dry weight of the plant material) and is of too low a molecular weight to directly substitute for Hevea or guayule NR. (However, sunflower NR has the consistency of rubber cement ("oily", resinous, and semi-fluid) and may be useful in certain elastomeric applications.)

It is unlikely that sunflowers could be bred to produce larger amounts of higher molecular weight NR because there does not appear to be enough genetic variability in this biosynthetic characteristic to justify or form the basis of a selection and breeding program. Thus, the economics of NR production from sunflowers do not compare favorably with production from domestic guayule plants, which are well known to yield 10-20% of a high molecular weight NR comparable to that of Hevea. In addition, because sunflower NR must be solvent-extracted from ground whole plant material (as in the case of guayule

R), it does not have a natural antioxidant (as does <u>Hevea</u> latex) and is nerefore highly susceptible to air oxidation. The need for the addition of antioxidant to preserve sunflower NR would thus add an additional cost to a lready unfavorable economic scenario. Therefore, because of the low uantities obtainable and the relatively poor qualities of sunflower NR, it is ighly unlikely that sunflower species could realistically serve as a omestic source of NR, even in times of national emergency, in the foreseeable uture.

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